

GERMAN DEMOCRATIC REPUBLIC

PATENT SPECIFICATION



(12) Commercial Patent

(19) **DD** (11) **224 341 A1**

Granted pursuant to Section 17 Para. Patent Law

4(51) C 30 B 25/02
H 01 L 21/205

PATENT AND INVENTION OFFICE

Published in the version submitted by the applicant

(21)	WPC30B/256098 0	(22)	10-28-1983	(44)	07-03-1985
------	-----------------	------	------------	------	------------

(71)	Akademie der Wissenschaften der DDR, 1199 Berlin, Rudower Chaussee 5, DD
(72)	Baranow, Boris, Dr. rer. nat. Dipl.-Chem., SU

(54) **Method for the separation of GaN and $Ga_xAl_{1-x}N$ epi layers from sapphire substrate**

(57) The objective of the invention is to allow multiple use of the hetero-epitaxial growth surface of the substrate, to prevent the destruction of the epitaxial layer by tensions between the substrate and the epitaxial layer, and to grow epitaxial layers for component manufacturing and physical analysis. Its task is to provide a method by which the epitaxial layer can be deposited removably from the substrate. The problem is solved by applying a thin layer of boron nitride to the substrate, onto which the active GaN or $Ga_xAl_{1-x}N$ epitaxial layer is deposited.

ISSN 0433-8481

5 pages

BEST AVAILABLE COPY

- 1 -

Method for the separation of epitaxial GaN and $\text{Ga}_x\text{Al}_{1-x}\text{N}$ Layers from Sapphire Substrate

Field of Application

This method is used to make gallium nitride or gallium aluminum nitride epi layers removable from the sapphire substrate. It can be applied to the manufacture of gallium nitride or gallium aluminum nitride-based opto-electronic components.

Characteristics of known engineering solutions

Due to their wide band gap, gallium nitride and gallium aluminum nitride are used as materials for electro-luminescent components. The preferred method of depositing gallium nitride and gallium aluminum nitride layers onto heated substrates is the process of vapor-phase epitaxy described in DD-WP 136 456 and for gallium nitride layers described in DE-OS 24 00 163. Preferred substrates for epitaxial deposits are sapphire disks. The crystallographic perfection of the epitaxial layer is determined by the surface quality of the substrate. This means that the substrates themselves have to be prepared very carefully, since the perfection of the epitaxial layer determines the quality of the component. The substrates, especially in hetero-epitaxy, are not involved in the actual function of the semiconductor component. Tensions between the substrate and the epitaxial layer may also lead to the destruction of the active layer.

- 2 -

Objective of the Invention

The invention has the objective to allow multiple use of the substrates for hetero-epitaxy through removal of the deposit layer from the substrate, to prevent the destruction of the epitaxial layer due to tensions between the substrate and the epitaxial layer, and to provide carrier-free epitaxial layers for the manufacturing of semiconductors and for physical tests, e.g for diagnostics of the layer material by way of absorption spectroscopy, luminescence measurements, or piezoelectric and electrical tests with contacts using the sandwich design.

Characteristics of the Invention

The invention has the engineering task to provide a method by which gallium nitride or gallium aluminum nitride layers having been deposited by vapor-phase epitaxy onto a sapphire substrate can be removed from the substrate in a non-destructive way. This method should preferably become an integrated process step of component manufacturing.

The invention solves this problem by proposing that a thin layer (some 10 Å) of hexagonal boron nitride is initially applied to the surface of the substrate via vapor-phase epitaxy. Using known methods, the gallium nitride or gallium aluminum nitride layer is then epitaxially deposited onto this thin layer.

The thin layer of boron nitride does not affect the substrate's orientation effect on the epitaxial layer, but reduces the substrate's wettability. At the end of the epitaxial process the different heat expansion factors lead to the separation of the epitaxial layer from the substrate's surface. The epitaxial layer lies freely on the surface of the substrate, and can be processed into a component, while the sapphire substrate can be used for more deposits. The method described in the invention can be integrated into the normal manufacturing process, immediately prior to the epitaxial deposit of the active layer.

- 3 -

In order to produce the boron nitride layer, the sapphire substrate - after having been cleaned according to the traditional process - is heated to 1050 °C, and an $\text{NH}_3\text{-BCl}_3$ -mixture is introduced into the recipient. The time required for exposure depends on the structure of the recipient and the concentration of the $\text{NH}_3\text{-BCl}_3$ -mixture, and takes a few seconds. If the BN-layer is too weak, the epitaxial layer to be deposited onto this BN-layer in the next process step will not separate from the substrate; if the BN-layer is too strong, the orientation effect of the substrate will be compromised. Under optimum conditions, the separated GaN or $\text{Ga}_x\text{Al}_{1-x}\text{N}$ layer has an equally smooth, mirror-like surface as the substrate.

Embodiment

In order to create a self-supporting, carrier-free GaN or $\text{Ga}_x\text{Al}_{1-x}\text{N}$ layer, a sapphire substrate wafer in a recipient was heated to 1050 °C, and a $\text{NH}_3\text{-BCl}_3$ -mixture with excess NH_3 was introduced inside the nitrogen flow for 10 seconds. Afterwards, following the instructions specified in DD-WP 136 456, the GaN and the $\text{Ga}_x\text{Al}_{1-x}\text{N}$ layer was deposited on the heated substrate out of an $\text{NH}_3\text{-GaCl}_3$ and $\text{NH}_3\text{-GaCl}_3\text{-AlCl}_3$ -mixture with low NH_3 partial pressure. After achieving the desired thickness of the layer, the supply of reactants and the heating of the substrate were stopped. The substrate was cooled in the carrier gas stream. As a result, the epitaxial layer separated from the substrate and rested freely on it. The carrier-free epitaxial layer had the same mirror-like surface as the substrate.

Claims

1. Method for the separation of epitaxial GaN or $\text{Ga}_x\text{Al}_{1-x}\text{N}$ layers from sapphire substrate, wherein a boron nitride layer with the thickness of some 10 Å is deposited onto the substrate upon which the GaN or $\text{Ga}_x\text{Al}_{1-x}\text{N}$ layer is deposited.
2. Method according to 1, wherein the boron nitride layer is deposited out of an $\text{NH}_3\text{-BCl}_3$ -mixture via vapor-phase epitaxy.

**EXPERT TRANSLATION BUREAU, INC.**

920 W. Lakeside, Suite 2109, Chicago, IL 60640
Telephone: (773) 271-5500 Facsimile: (773) 728-1370

www.Expert-Translation.com

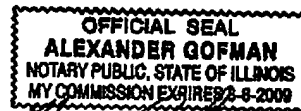
CERTIFICATE OF TRANSLATION

March 31, 2005

I, Angela Christie, hereby certify that I am competent in German and English languages. I further certify that under penalty of perjury translation of the aforementioned patent application from the German language into the English language is accurate and correct to the best of my knowledge and proficiency.

Angela B. Christie

Professional Translator



Alex Gofman
03.31.2005

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ BLACK BORDERS
- ☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
- ☐ FADED TEXT OR DRAWING
- ☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
- ☐ SKEWED/SLANTED IMAGES
- ☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
- ☒ GRAY SCALE DOCUMENTS
- ☐ LINES OR MARKS ON ORIGINAL DOCUMENT
- ☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
- ☐ OTHER: _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.